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Indian Standard

METHOD FOR ESTIMATION OF CARBOXYLIC ACID GROUPS IN CELLULOSIC TEXTILE MATERIALS

PART II SODIUM CHLORIDE-SODIUM BICARBONATE METHOD

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METHOD FOR ESTIMATION OF CARBOXYLIC ACID GROUPS IN CELLULOSIC TEXTILE MATERIALS

PART II SODIUM CHLORIDE-SODIUM BICARBONATE METHOD

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Indian Standard

METHOD FOR ESTIMATION OF CARBOXYLIC ACID GROUPS IN CELLULOSIC TEXTILE MATERIALS

PART II SODIUM CHLORIDE-SODIUM BICARBONATE METHOD

0. FOREWORD

0.1 This Indian Standard (Part II) was adopted by the Indian Standards Institution on 4 October 1974, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

0.2 In the cellulosic textile industry, cellulose in the form of fibres, yarn and fabric comes in contact with different oxidizing agents during the various chemical processing treatments. The action of these oxidizing agents on cellulose may result in the formation of oxycelluloses of acidic character attributable to the introduction of carboxyl groups into the cellulose chain molecule. Purified cotton cellulose, not subjected to any treatment with oxidizing agents, also behaves as though it possesses a very small content of carboxylic acid groups. The absorption of metallic ions from aqueous solutions of their salts is an outstanding property of oxycelluloses. This property in celluloses and oxycelluloses is due to the presence of carboxylic acid groups in them. The estimation of carboxylic acid groups present in cellulosic textile materials is a method of determining the extent of this type of oxidation of cellulose. This is a useful supplementary test to other tests such as copper number and fluidity tests.

0.3 The iodometric method for the estimation of carboxylic acid groups in cellulosic textile materials is covered in Part I of the standard.

0.4 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part II) prescribes the sodium chloride-sodium bicarbonate method for estimation of carboxyl acid group content of cellulosic textile materials.

*Rules for rounding off numerical values (*revised*).

2. PRINCIPLE

2.1 Sample is de-ashed with hydrochloric acid, washed, soaked in sodium chloride-sodium bicarbonate solution and then filtered off. An aliquot of the filtrate is titrated with 0.01 N hydrochloric acid to a methyl red end point. The amount of the sodium chloride-sodium bicarbonate solution consumed is a measure of the ion-exchange capacity of the cellulose and indicates the extent of carboxyl content.

3. SAMPLING

3.1 Sample shall be so drawn as to be representative of the lot.

3.2 Sample drawn in accordance with the procedure laid down in the relevant material specification or in compliance with an agreement between the buyer and the seller shall be held to be representative of the lot.

4. PREPARATION OF TEST SPECIMENS

4.1 Condition the sample under test in the prevailing atmosphere for at least 20 minutes and cut it into small pieces. Mix all the pieces thoroughly and draw at least 2 test specimens, each weighing about 2.5 g. At the same time draw specimens for moisture content determination.

5. APPARATUS

5.1 Fritted Glass Funnels

5.2 Erlenmeyer Flasks — of Pyrex glass (or similar heat-resistant glass), fitted with a glass stopper and of 250 ml capacity.

6. REAGENTS

6.1 Quality of Reagents — Unless specified otherwise, pure chemicals shall be employed in the test and distilled water (see IS : 1070-1960*) shall be used where the use of water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

6.2 The reagents required for the test shall be as given below.

6.2.1 Hydrochloric Acid — 0.01 N.

6.2.2 Hydrochloric Acid — 1 : 99. Dilute 1 volume of concentrated hydrochloric acid (sp gr 1.19) with 99 volumes of water.

6.2.3 Methyl Red Indicator Solution

6.2.4 Sodium Chloride — Sodium Bicarbonate Solution — Dissolve 5.85 g of sodium chloride and 0.84 g of sodium bicarbonate in water and dilute to 1 litre.

*Specification for water, distilled quality (*revised*). (Since revised).

6.2.5 Sodium Hydroxide Solution — 0.4 g/l.

6.2.6 Water Saturated with Carbon Dioxide.

7. PROCEDURE

7.1 Weigh one test specimen accurately, disintegrate it in water and filter through a fritted glass funnel. Disperse the disintegrated specimen to about 1 percent consistency in hydrochloric acid (1 : 99) at room temperature. After 2 hours collect the specimen on a fritted glass funnel and wash with water saturated with carbon dioxide. Continue washing until the filtrate, after boiling does not require more than two drops of sodium hydroxide solution to give an alkaline colour (yellow) with methyl red.

7.2 Weigh the wet pulp pad, transfer it to an Erlenmeyer flask, add 50 ml of the sodium chloride-sodium bicarbonate solution with a pipette and shake to obtain a homogeneous slurry (see Note). Allow the mixture to stand for 1 hour at room temperature. Filter through a clean, dry fritted glass funnel. Pipette 25 ml aliquot of the filtrate into an Erlenmeyer flask and titrate with 0.01 N hydrochloric acid using methyl red indicator. When the first change in colour occurs, boil the solution for about 1 minute to expel carbon dioxide and continue the titration to a sharp end point.

NOTE — If the cation exchange capacity is very low, use a solution containing about 5.85 g of sodium chloride and 0.42 g of sodium bicarbonate per litre. It is important that the excess of sodium bicarbonate should be large enough so that the pH does not fall below 7.0.

7.3 Blank — Pipette 25 ml of sodium chloride-sodium bicarbonate solution into an Erlenmeyer flask and titrate as in 7.2.

7.4 Determine the moisture content of the sample using the specimens taken for the purpose (see 4.1) as given in IS : 199-1973* and calculate the oven-dry mass of the specimen taken for the test (see 7.1).

7.5 Similarly test other test specimen(s).

8. CALCULATION

8.1 Calculate the carboxylic acid group content of each specimen, in milli-equivalents of—COOH per 100 g of the specimen, by the following formula :

$$\begin{aligned} &\text{Carboxylic acid group content, as} \\ &\text{milli-equivalents of—COOH per 100 g} \\ &\text{of specimen} = \left(V_1 - V_2 - \frac{V_3 m}{50} \right) \frac{2}{M} \end{aligned}$$

where

V_1 = quantity in millilitres of 0.01 N hydrochloric acid consumed in the blank (see 7.3),

*Methods for estimation of moisture, total size or finish, ash and fatty matter in grey and finished cotton textile materials (second revision).

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V_2 = quantity in millilitres of 0.01 N hydrochloric acid consumed
in the actual test (see 7.2),

m = mass in grams of water in the wet pulp pad, and

M = oven-dry mass in grams of test specimen (see 7.4).

8.2 Calculate the average of the values obtained as in 8.1.

9. REPORT

9.1 The report shall include the following:

- a) Carboxylic acid group content, and
- b) Number of specimens tested.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Conversion
Force	newton	N	1 N = 0.101 972 kgf
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²

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